SYNOPSIS

Investigation of Structural, Electrical, Magnetic and Optical Properties of Mixed Manganese Ferrite Nanoparticles

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1 INTRODUCTION

Nanotechnology refers to the technology of materials having dimensions in nano range. Existence of nano particles is not new. In earlier times, there were several artifacts which were made of nanoparticles. People used nanoparticles in various fields like art, medicine, photography and others because of their advantageous properties. Amongst a variety of ferromagnetic materials, ferrites are the one having iron (III) oxide as the chief ingredient. The research on ferrites has its roots in the early part of the last century. The major role to explore the ferrites from science & technological point of view is in the lap of Philips researchers at Netherlands, especially the pioneer, J. L. Snoek who visualized the significance of ferrites in the telecommunication industry [1]. In recent years, ferrites have attracted materials scientists owing to their fascinating electric and magnetic properties at the nanoscale for the industrial & medical applications.

1.1 PROPERTIES OF FERRITES

(i) **Excellent chemical stability:** It is the advantageous property of material which counters the change in material due to any internal or external factors. The ferrites possess good chemical stability which allows their applications in industry.

(ii) **High Curie temperature:** Curie temperature is the temperature above which a ferromagnetic substance starts behaving as a paramagnetic substance. Ferrites have the higher values of Curie temperature from about 400 to 500K.

(iii) **High resistivity:** Resistivity is a property of material which represents the opposition of material to the flow of electric current. High values of resistivity in ferrites indicate their insulation properties. Therefore, ferrites are good insulators.

(iv) **Permanent Magnetism:** The ferrites are ferromagnetic materials therefore they possess magnetization value which is even present when there is no magnetic field. Hence these are good source to make permanent magnets.

(v) **High value of Permeability:** Permeability is the measure of ability of material to allow the formation of magnetic field within itself. Ferrites generally have a higher value of permeability which is used in various magnetic applications.

1.2 APPLICATIONS OF FERRITES

(i) **Transformer cores:** Soft ferrites with low value of coercivity are used in making transformer cores as their winding is made up of ferrites. Resistivity of

ferrites acts as insulator in transformer giving more resistance to higher values of currents. It also helps to prevent eddy currents [2].

(ii) **Memory cores of computers:** Before, the discovery of semiconductor memory chips Ferrites were the major form of electronic memory used in computers. This electronic memory component consisted of several tiny rings known as cores which are made of a magnetic ferrite material [2].

(iii) **Noise filters:** Most of the noise suppression components make use of capacitors, and these work by discharging the noise to the ground, therefore they are useless without a stable ground. On contrary, ferrite cores can suppress noise without grounding and hence these are helpful in noise filtration [3].

(iv) **Satellite communication:** Ferrites are used in satellite communication to control the radiation by applying a thin layer of ferrite as superstrate or random layer. It not only control the radiation but also help in reception and scattering form a printed array or antenna by applying a dc magnetic bias field in the plane of the ferrite, which is perpendicular to the RF magnetic field [4].

(v) **Radio receivers:** The ferrite bar antenna is a form of RF antenna that is mostly used in portable transistors broadcast receivers and many hi-fi tuners worldwide. This is done by wounding a coil around the ferrite rod and connecting a variable capacitor to make resonance [5].

1.3 CLASSIFICATION OF FERRITES

There are two broad categories on the basis of which ferrites can be classified. The first one is soft ferrites which are known to have low coercivity and are easy to magnetize as well as demagnetize while the second is hard ferrites which are known to have high coercivity.

Table 1 Types of ferrites with structure, chemical formula and examples

Type	Structure	General Formula	Example
Spinel	Cubic	MFe_2O_4	M = Mn, Zn, Ni, Mg, Cu, Co, Li, Cd etc.
Garnet	Cubic	$Ln_3Fe_5O_{12}$	Ln = Y, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Lu etc.
Magnetoplumbite	Hexagonal	$MFe_{12}O_{19}$	M = Ba, Sr
Orthoferrites	Pervoskite	$LnFeO_3$	Ln = same as in Garnet

On the other hand, by considering the crystal structure and magnetic ordering into account, there are four major categories into which ferrites can be classified. Table 1 represents the four categories of the ferrites based on the crystal structure [6].

1.4 STRUCTURE OF SPINEL FERRITES

The chemical representation of the ferrites having spinel structure can be represented as $(M^{2+})[Fe_2^{3+}]O_4$, where M^{2+} represent divalent metal ions or the mixture of divalent ions. The schematic picture of spinel structure is shown in Fig. 1 [7].

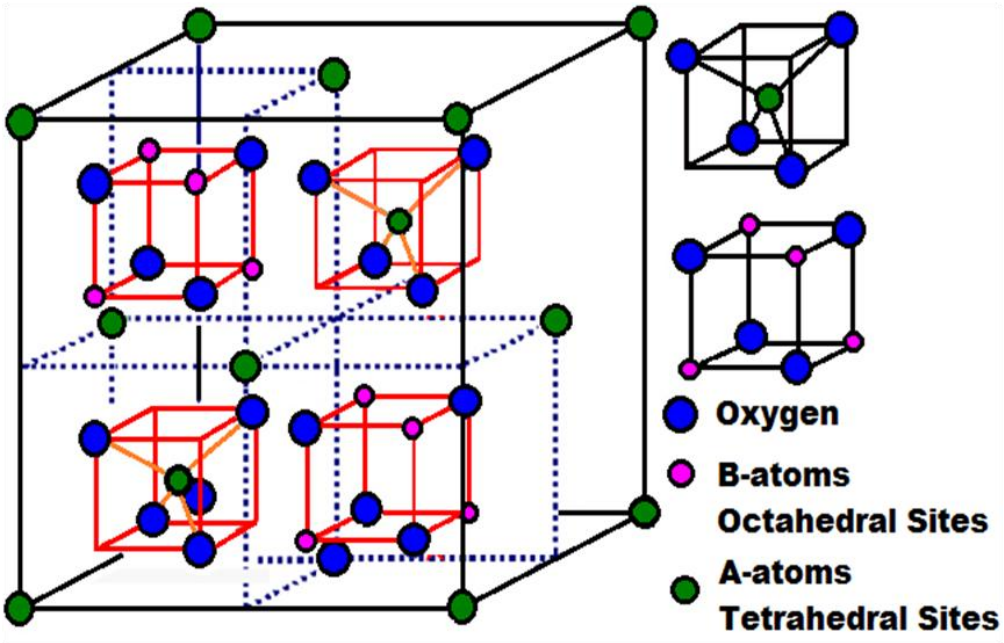


Fig. 1 Schematic picture of spinel structure

In a unit cell of spinel ferrite, there are 8 formula units i.e. ($8 \times MFe_2O_4$). Therefore, a unit cell consists of 8 divalent metal ions, 16 ferric ions and 32 oxygen ions [8]. The $32O^{2-}$ ions are known to outline a closed packed FCC arrangement which consists of 2 types of interstitial sites. One of interstitial site is recognized as tetrahedral site usually referred as A-site. This site is bounded by 4 oxygen ions. Second interstitial site is recognized as octahedral site usually referred as B-site. This site is bounded by six oxygen ions. In spinel ferrites, there are sixty four A-sites and 32 B-sites. Out of 64 A-sites and thirty two B-sites cations only engage 8 A-sites and 6 B-sites respectively [9]. In spinel ferrites, the general division of metal ions over tetrahedral site and octahedral site can be symbolized as $(M_{1-x}^{2+}Fe_x^{3+})[M_x^{2+}Fe_{2-x}^{3+}]O_4^{2-}$, where parenthesis indicates the cations present at A-site while the square bracket indicates the cations present at B-site. The symbol 'x' is known as the inversion parameter and on the basis of inversion parameter; the spinel ferrites can further be categorized in three types. For x = 0, the ferrites are acknowledged to comprise normal spinel structure, in support of x = 1, ferrites are acknowledged to comprise inverse spinel structure and in support of $0 < x < 1$, ferrite are known to have mixed ferrites. Thus, the proficiency to allocate the metal ions on A-site and B-site create ferrites extremely appealing materials where a

material scientist can play with the properties but this distribution influenced by many factors such as magnitude of the ionic radii, electronic configuration of the metal ions and electrostatic energy of the spinel lattice [10-11]

2 LITERATURE REVIEW

Desai et. al [12] in 2019 have synthesized manganese ferrites by combustion method and Polyvinyl alcohol was added to the mixed metal salt solution. The resulting particles were characterized by X-ray diffraction , X-ray photoelectron spectroscopy , scanning electron microscope, tunnelling electron microscope and Ultraviolet–Visible spectroscopy properties. The carbon content analysis showed a slight increase in the percentage of carbon as PVA content increased. However, the carbon content was always less than 0.2% by weight. No hysteresis has been found at 300 K for the catalysts. Such behavior is attributed to the formation of ferro/ferri-magnetic nanoparticles.

Arshad et. al [13] in 2019 have synthesized cobalt and nickel doped manganese ferrites nanoparticles by co-precipitation method and reported the structural, magnetic and dielectric properties. From XRD it has been reported that all samples possessed cubic spinel structure. The crystallite size lies in the range of 27–35 nm. The spectral analysis confirmed bending and stretching of bonds at octahedral (B) and tetrahedral (A) sites. The spectral bands were shifted towards the longer wavelength side and these indicate the incorporation of dopants cations (Co-Ni). From M-H loops of nanoferrites it has been found that with the increase of Co-Ni contents, the saturation magnetization increased up to $x = 0.15$ and then it decreased.

Kafshgari et. al [14] in 2019 have synthesized manganese ferrites by three different methods including the co-precipitation, sol-gel, and hydrothermal route and reported structural and magnetic properties. From Debye- Scherrer's formula peaks indicated that the mean size of manganese ferrites nanocrystallites obtained to be 36nm for co-precipitation, 45nm for sol-gel and 16nm for hydrothermal. Field emission scanning electron microscopy analysis images confirmed the existence of three types of $MnFe_2O_4$ nanoparticles: spherical shape, multi-walled hollow nanosheets, and reticular structure. These findings show that various methods of nanoparticles synthesis can lead to different particle sizes and magnetic properties.

Muntean E et. al [15] in 2018 have synthesized manganese ferrites/ carbon composites with different PVA content by thermal combustion method and studied structural and magnetic properties with variable content of carbon. Fourier-transform infrared spectroscopy confirmed the redox interaction between PVA and nitrate iond at around 100^0C. The saturation magnetization decreased with the increase of carbon amount from 47.9 emu g^{-1} to 18.5 emu $g^{-1}$and was correlated with PVA content.

Jacintha et. al [16] in 2017 have synthesized nanosized manganese ferrites by sol-gel method using natural polymers like wheat flour (WF) and potato flour as surfactants and its structural, morphological, optical and magnetic characteristics were studied. Mean size of manganese ferrites nanocrystallites has been reported 23nm for WF/$MnFe_2O_4$ and 16 nm for PF/$MnFe_2O_4$.

Bashir et. al [17] in 2017 have fabricated graphene nanosheets with copper doped manganese ferrite nanoparticles and reported electrical properties. It has been observed that electrical conductivity of manganese ferrites was enhanced by doping of conducting metal copper ions.

Oleveira et. al [18] in 2016 synthesized mixed manganese-copper ferrites and investigated for their electric and magnetic properties. From X-ray analysis lattice parameter increases linearly with increase in Mn content, due to ion substitution. In study the resistivity is found to be decreased with increasing temperature for all samples. The ferrites showed semiconducting behaviour has been reported. Electron transport in the investigated samples is dominated by grain/barrier model adapted to this crystallite scenario at high temperatures (T=423K).

Salah et. al [19] in 2016 have synthesized copper doped manganese ferrites by co-precipitation method and studied structural and magnetic properties. The lattice parameters decrease leading to the increase in X-ray density with increasing the copper concentration. The decrease in the crystallite size with increasing the copper content is attributed to the higher formation temperature. The AC conductivity increases with increasing copper concentration. The dielectric constant and dielectric loss showed a decrease with increasing frequency and increase with increasing temperature for all compositions.

Kaiwen et. al [20] in 2015 have synthesized manganese-nickel ferrites with lithium substitution by precursor method and reported structural, morphological,

optical and magnetic characteristics. Manganese-nickel ferrites with lithium substitution had highest specific saturation value 57.94 emu g^{-1}.

Zipare Kisan et. al [21] in 2015 have synthesized manganese ferrite using chemical co-precipitation method and characterized for its structural and magnetic properties. From X-ray diffraction pattern lattice parameter 8.5075A^0 has reported. From TEM average size of particle has reported 5nm. The magnetic measurements reported super paramagnetic nature with saturation magnetization 69emu g^{-1}.

Zeng et. al [22] in 2015 have synthesized manganese ferrites by solvothermal method and reported electromagnetic properties. The permittivity dispersion behavior is explained by Debye dipolar relation expression and complex permeability is analyzed using Landau- Lifshitz-Gilbert equation. Eddy current loss arises at different frequencies has reported.

Kishore et. al [23] in 2014 have synthesized nanocrystalline mixed ferrites by chemical co-precipitation route. They have reported structural and electrical properties. Dielectric measurements were performed within frequency window of 1 Hz to 1 MHz and explained dielectric dispersion.

Goswami et. al [24] in 2013 have synthesized manganese ferrites nanoparticles by acetates precursor method and reported magnetic properties. Magnetization of ferrites particles increases with calcinations temperature. Calcination at 950 ^oC leads to formation of rods with grain growth that introduces large shape anisotropy has been reported. The magnetization curve for rods does not reach saturation and paramagnetic behavior reported.

Sundari et. al [25] in 2013 have applied a citric acid anionic surfactant for nano manganese ferrites using sol gel method and reported structural change when calcinations have been varied. The UVDR analysis reported a high absorptive band gap after 4oo nm for 600 ^0C calcinated manganese nano ferrites.

Mostafa et. al [26] in 2013 have synthesized cadmium substituted manganese ferrites nanoparticles and reported structural and magnetic properties. For $Mn_{1-x}Cd_xFe_2O_4$ powder in XRD data showed pure single phases of cubic with x upto 0.3. However sample with $x \geq 0.5$ showed hexagonal phase of cadmium hydroxide besides the ferrite phase.

Gunjal et. al [27] in 2012 have synthesized chromium substituted manganese ferrites by the wet chemical co-preparation method. They reported structural and

electrical properties. The X-ray diffraction result showed that system exhibit a single phase with cubic spinel structure. Lattice constant decreased linearly with Chromium concentration due to small ionic radius has been reported. Dielectric constant and dielectric loss tangent decreases with increase frequency and reported strong influence of Chromium substitution.

Farooq et. al [28] in 2012 have synthesized manganese ferrites nanoparticle via co precipitation route and reported structural and electrical properties. Dielectric constant and capacitance were studied at room temperature in the frequency range of 600 Hz to 1 MHz by LCR meter which showed exponential decay at low frequency for both while both becomes independent of frequency in higher range.

Naseri et. al [29] in 2011 have synthesized manganese ferrite nanoparticles by thermal treatment method and reported structural and magnetic properties. The average particle size of manganese ferrites were determined by TEM, which increased with the calcinations temperature from 12 nm to 22 nm reported. Super magnetic behavior for all samples and saturation magnetization increases from 3.06 to 15.78 emu/g by increasing calcinations temperature has been reported.

Bolarín-Miró et. al [30] in 2011 have synthesized nanocrystalline manganese ferrites by mechanosynthesis and reported magnetic properties. Manganese ferrite did not form when MnO_2 was used but Mn_2O_3 formed manganese ferrite. The maximum saturation magnetization value reported 49.77 emu/g.

Lazarević et. al [31] in 2011 have synthesized manganese ferrites via sift mechanochemical route and reported surface morphology, magnetic properties and have done Mössbauer measurements. Two cases studied: (a) $Mn(OH)_2$ and Fe_2O_3 and (b) $Mn(OH)_2$ and $Fe(OH)_3$. Crystallite size of about 40 nm and 50 nm has reported amd studied magnetization value of 70.4 emu/g and 71.1 emu/g respectively.

Yang et. al [32] in 2010 have synthesized water-soluble super paramagnetic manganese ferrite nanoparticles for magnetic resonance imaging applications. They formed nanoparticles of diameter of 7nm and reported super paramagnetic with saturated magnetization of 39 emu/g.

Ashiq et. al [33] in 2009 have synthesized nanosized cobalt manganese ferrites via co-precipitation route and reported structural, electrical and magnetic properties. XRD data showed samples of single phase and crystallite size is found to be in range of16-18 nm. They reported electric resistivity of samples is found

to increase from 0.22×10^7 to 14.5×10^7 ohm cm and activation energy from 0.34 to 0.40 eV. They reported increase in saturation magnetization from 18.33 to 50.63 emu/g X=0.4 and then decreases.

Deraz et.al [34] in 2009 they have synthesized single-domain manganese ferrites by combustion technique and reported structural and magnetic properties. They have reported maximum magnetization and coercivity values 68.58 emu/g and 62.57 Oe respectively.

Lakshman et.al [35] in 2006 have prepared chromium ions substituted magnesium copper manganese ferrites and reported structural and magnetic properties. The single phase spinel structure of these samples has been confirmed from XRD. Mossbauer spectra of these samples studied at room temperature reported two characteristics ferromagnetic Zeeman sextets for lower concentration of Cr^{3+} followed by relaxation phenomenon and spectrum collapses to a paramagnetic doublets.

Li. Y et. al [36] in 2004 have prepared mixed manganese ferrites and reported structural properties.

Rana et. al [37] in 1999 synthesized copper substituted manganese ferrites by standard ceramic method and reported relationship between structural parameters and copper substituted ions.

3 RESEARCH GAP IDENTIFICATION

As we know the substitution plays a critical role in the synthesis of ferrites for a particular application, therefore, many researchers have investigated substituted manganese ferrites in the bulk as well as in the nano dimensions. According to literature survey there is no report available on the structural, electric, magnetic and optical properties of the copper and aluminium substituted manganese nanoferrites by solution combustion method. Further, there are several reports which suggested that the method of synthesis plays a crucial role in the final properties of the ferrites. Therefore, in the present work we aimed to synthesize mixed manganese nanoferrites by solution combustion technique and to investigate the structural, electrical, magnetic and optical properties of copper and aluminium substituted manganese nanoferrites.

4 OBJECTIVES OF THE PRESENT WORK

In fact there is enormous scope to explore the manganese nanoferrites but the main objectives of this study are:

(i) To prepare the series of copper and aluminium doped manganese nanoferrites by solution combustion technique of the compositions

(a) $MnCu_xFe_{2-x}O_4$ (x = 0.0, 0.1, 0.2 and 0.3)

(b) $MnAl_yFe_{2-y}O_4$ (y = 0.0, 0.1, 0.2 and 0.3)

(ii) To study the structural and morphological properties of copper and aluminum doped manganese nanoferrites.

(iii) To study the electrical, magnetic and optical properties of copper and aluminum doped manganese nanoferrites.

5 PROPOSED OUTCOMES OF THE PRESENT WORK

(i) The present work shall be beneficial to understand that a low cost method i.e. solution combustion technique can be employed to synthesize the ferrites at nanoscale.

(ii) The structural parameters which shall be investigated in this work shall be beneficial to understand the structural modifications made by the substitution in the manganese nanoferrites.

(iii) The electrical study shall be beneficial to understand the impact of substitution on the dielectric constant and dielectric loss tangent of the mixed manganese nanoferrites. Further, the study shall be beneficial to understand the conduction theories involved in the various experimental results.

(iv) The magnetic study shall be beneficial to understand the effect of substitution on the exchange interactions in manganese nanoferrites while the optical study shall be beneficial to understand the effect of substitution on the band gap and metal oxygen bonds in manganese nanoferrites.

6 PROPOSED METHODOLOGY:

(i) Synthesis of copper and aluminium substituted manganese nanoferrites: the samples shall be synthesized via solution combustion method. For this, in a stoichiometric ratio ferric nitrate, copper nitrate, manganese nitrate, and glycine will be mixed in 80 ml distilled water to obtain the precursor solution. The obtained precursor solution shall be heated at optimized temperature with constant stirring. The obtained powders will be pre-sintered at optimized temperature so as to obtain the composites. Sintered powders will be hydraulically pressed so as to obtain the pellets. Prepared pellets will be sintered at optimized temperature and will be subsequently cooled to room temperature.

Pellets will be coated with silver paste to make electric contact for electrical measurements. This procedure shall also be opted for the synthesis of aluminium substituted manganese nanoferrites series.

(ii) Structural and morphological study: The structural study of the synthesized samples shall be carried out by the X-ray diffraction study which will be made by Cu-K_α radiation of wavelength λ=1.54 Å. The morphology of the synthesized samples shall be carried out by Field Emitting Scanning Electron Microscopy.

(iii) Electric and dielectric study: The dielectric constant and dielectric loss tangent will be investigated as a function of frequency and substitution.

(iv) Raman spectroscopic and FTIR study: The Raman spectroscopic study shall be carried out to study Raman vibration frequency bands and FTIR study shall be carried out to investigate the effect of substitution on metal oxygen bonds.

(v) Magnetic study: The hysteresis study of the synthesized samples shall be carried out at room temperature. The magnetization technique shall be used to verify the distribution of cations obtained by employing x-ray diffraction method.

7 MEASUREMENTS COMPLETED OR OBJECTIVES ACHIEVED

(i) Synthesis of copper and aluminium substituted manganese nanoferrites: Copper and aluminium substituted manganese nanoferrites have been synthesized by solution combustion method.

(ii) Structural and Morphological study: X-ray diffraction study of copper doped series has been done. Structural parameters like particle size, lattice parameter, x-ray density and hopping lengths have been calculated. The surface morphology of the copper substituted manganese nanoferrites have been studied by using scanning electron microscopy.

(iii) Raman spectroscopic and FTIR study: FTIR study has been carried out for copper doped series.

(iv) Magnetic study: The room temperature hysteresis study of copper doped series has been carried out and magnetic parameters like saturation magnetization, coercivity and retentivity has been estimated from the obtained results.

8 THESIS OUTLINE:

Thesis shall comprise four chapters as:

Chapter 1 (Introduction): This chapter includes the introduction of ferrites & types of ferrites, literature review, reasons for the doping of copper and aluminum in Manganese nanoferrites. The chapter shall also include magnetism and electrical related theories etc.

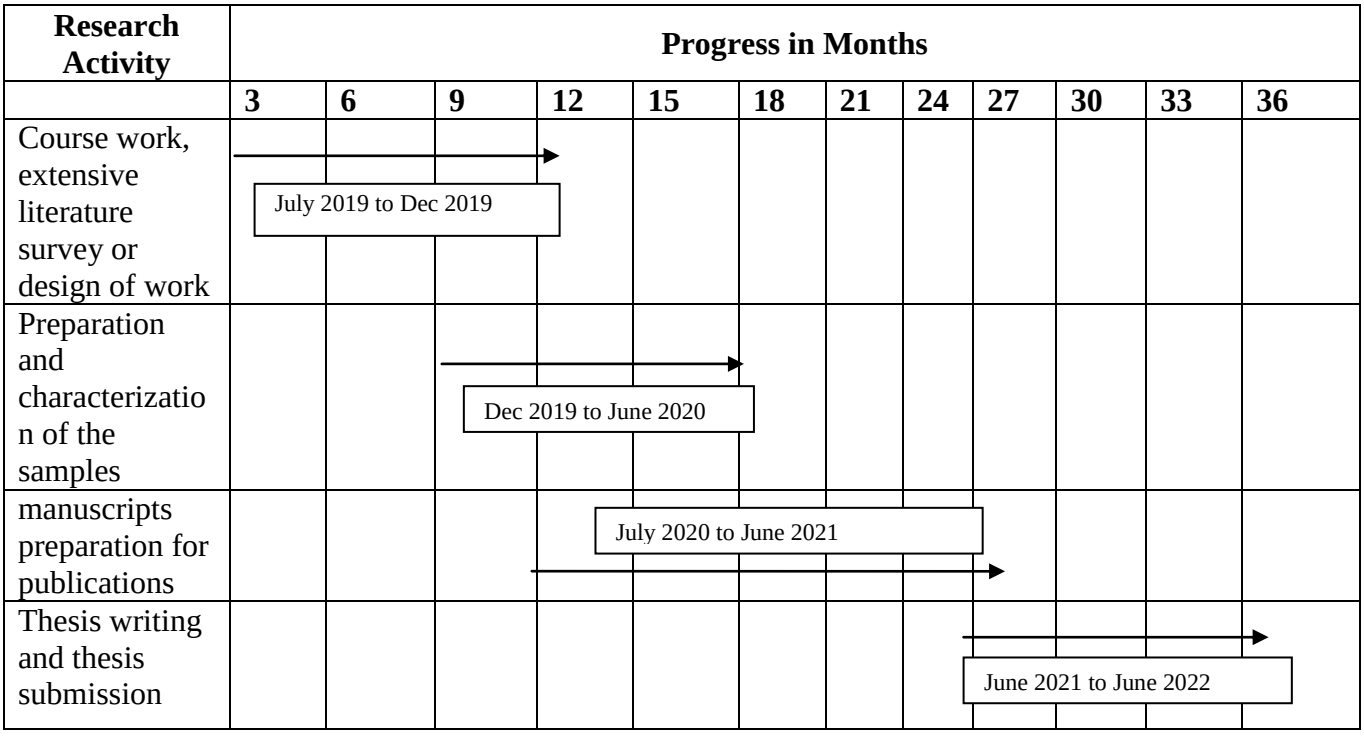
Chapter 2 (Experimental and Characterization Techniques): It includes the details of the used synthesis method and instrumental details. Further, the chapter shall include the principle/theories on which some used instruments work.

Chapter 3 (Structural Study): This chapter shall deals with the results of structural parameters obtained and explanations to the obtained results.

Chapter 4 (Electrical, Magnetic and Optical Study): This chapter shall deals with the results of electrical, magnetic and optical properties and explanations to the obtained results.

Summery and Conclusion: This chapter will Comprises summary of entire study according to objectives along with conclusions. The scope of further research on the basis of recommendations shall also be included in this chapter.

8 Research Plan Schedule:



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